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TITLE: Method and apparatus for detecting hydrogen cyanide

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Patented Dec. 19, 1950 2)534,229 @@UNITE'D STATES PAT,ENT @OFFICE 2,534,229 METHOD-AND-APPARATUS@FORIDETEC,T)ING. @HYDROGEN CYANIDE 'Homer W. Citrhart, and John A. Krynitsk @ Y, , Washington, D. C. Applicatioti'March'.15,1945, Serial No. 582,965 15- Cl@tiins. (Cl. 23-232) ,(Granted under the act of - Mareh 4 3, 1883@ as amended7@April 30, 1928,1 370 0.@ G@ 757) Our invention relates 1:@tol. the @ detecti6n,. of 'hYr@ dr-ogen- eya@nide or hydrocyanic, acid in; admixture with air or other gases: @ U-nder ordinary conditions, hydrocyanic . or prussic- @acid. i@a- a light gas haN7ing-an,@iodor@ re- 5 sembling:that:ofbitter-a-Imonds@ One lof@its,,best know-n properties @ is@ @its@ extreme toxicity @ and- for this -reason it is frequently- used@ as:@:a@ fumigat- ing agent. Another very common industrial ap- plication of cyanide, com@pounds is in electro-plat- 10 ing,-work where. solutions containing. the cyanide radical,.are- conventional. The.slig ht-hydrolysis and,,decomposition of@.these-.compounds irl solu- tion,,results in.thecontamination.of the.@a-tmos.. pher.e,,in.-.electro@-plating. room wit h-appreciable 15 concentra.tions - In, @addition to. the. i@ndustritil . uses , of cyanido com 6unds, hydrocyanic@ acid itself. has- considbr- . p able value@,as.,a.,war-,gas. In spite of it,s volatility and,. hence, its, lack,of persistency, -it is, very ef@. 20 fective.because of its. extreme. toxi,,city and lack of Isuch,aetion,as 1:@chrymation or., irritation. It is.7a.well-establishe.d fact..that ' many people can- not. detect the odor- of ..hydrocyanic acid. even when @it is present -in.. the, atmosphere in concen- 25 trations, far.. exceeding,, lethal.. concentrations. It,is,an object,.of..our..invention.to,provide.a simple.and.convenient method . of., detecting- con- tainination of the-atmosphere.or other gases -with trae,es- of . hyd@tocyanic acid in amounts below @,,0 lethal ' concentra6ns. It is a second object of our invention t6 provide a device which can -be used,. simply and easils, to detect small concentrgtions of hydrocyanic acid in small'samples@@of,air @or@other,@gas. 35 It is @another. @bbiect@of -@our-,Invention..to provide a @@ colorimetric- quz@littctive;: and @ Bemi@ quantita;tive test apparatus- and:a @ composition.l.fori the.i. detee- tion. 6f.,' hydr@ocyanic - acid @ ound estimation of,@@ @it8 concentration; in a.,contaminated@- atmosphere. 40 A: further object of @.our. inventionl,is, to @provide a. method -@of @ preserving @,indefinitely chemical@. re" agents which are subject.@.to@%@decomi>osition' by contact@ with oxygen.@ @Other objects and@.advantages..Of our.:invention 45 will, in, part,,be @obvious. and,@in:@part@ appear,here- inafter' I Our invention@ compri8es the, @composition, methodl,and ' apparatus for preserving an oxygensensiti,ve@chemical@and,:for@detecting.-hydrocyanic 50 acid%by- a@sharp colorimetric reaction.@.w.-hich will be descnbed @in dettbil,and illustrated @@iir the drawing.; accompnaying. this-. specification. @The droiiiing@represents@a@@@longitudinal,@eross- se6tion.through- a;,Itube comprisiig:@the appara-tus 55 of".our. @invention., A satisfactory colorimetric reaction must be one which is, first; extremely sensitive, second, one which gives a very@ sharp color change which is readily distinguished, and, third" one whir,,h Preferably takes iplace, -Gn, a.,white background. There. are a number of reactions of hydrocyanic acid which- will -give colbr, among which is the reaction@of hydrocyanic@acid,with alkaline sodium Picratetafrmiso- purpuricacid. Asatestforthe detection of hydrocyanic.acid, this reaction is not P_articularly, satisf aetory for the color change @involved is one in which yellow- sodium pierate is converted to a reddish--brown iso-purpuric, acid. I-hydrocyanic. acid@ can also be made to react with cupric sulfide. However,- the reaction is not a good colorimetric one for it @ involies the con" version of 'the@ bia-ek cupric -sulfide to; f orm.white cuprous- cyanide The-reaction,.of the cyanide radical@or hydrocyanic. acid with iron salts to-give ferri- ferrocyanide is,-very well known. I-lowerver, it is to 0 complicated for convenient field use. We- have discovered@ o@ method whereby@ the reaction of- hydroeyonic acid with a cupric saitorganic aromatic, amine@ reagent can be used for the@ qualitative,.and: semi-quantitative detection of. hydrocyanic acid. The reaction gives@ a sharp color change and possesses the, advantage of being, rapid, direct and quite specific for hydroeyonic acid. In the past the principal obstacle to@.the use.. of

this type of reaction, that is, the cupric-salt-organic aromatic amine reagent, for the detection of hydrocyanic acid was the instability of the various formulations. Short periods of exposure to traces of oxygen discolored the material so badly it was made useless for colorimetric work. We have discovered that a cupric salt-aromatic-amine combination which is highly sensitive to hydrocyanic acid and, consequently, can be used to detect micro-concentrations of hydrocyanic acid, can be stabilized and preserved so that the reagent once prepared and sealed into the apparatus will remain in operative condition for an indefinite period. In general we have found that organic aromatic amines: such as aniline, ortho-phenylenediamine, para-toluidine, para-phenetidine, diphenylamine, dimethylaniline, N-benzylaniline, phenyl- α -naphthylamine, -bis-(p-methylaminophenyl) methane, Michler's ketone, dibenzylaniline, 4,4'-tetramethyldiaminotriphenylmethane, 4,4'-hexamethyltriaminotriphenylmethane, p-diethylaminodiphenylmethane, diphenylbenzidine, and tetramethyl diaminodiphenylmethane-, (or bis-(p-dimethylaminophenyl)). Yi